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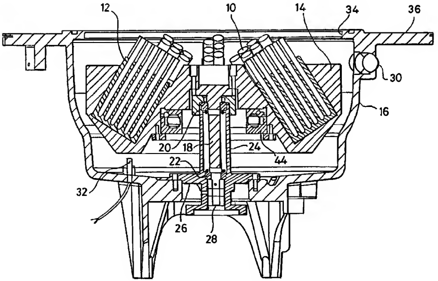
(54) Title: BALANCING DEVICE IN COMBINATION WITH CENTRIFUGAL EVAPORATOR




(57) Abstract: A rotor for use in a centrifugal evaporator is described, having a plurality of sample holder receiving means and fitted with an automatic balancing device for reducing out of balance forces that arise from the uneven distribution of sample holders around the rotor as the rotor is rotated during an evaporation process. A process of evaporating a plurality of samples is also described, in which sample holders containing samples are loaded initially and centrifuged, the rotor is stopped and some of the sample holders are removed and/or other sample holders are added before the rotor is spun again; wherein a force to compensate for different and uneven loading of the rotor by the samples and sample holders is generated by a balancing device which comprises a housing defining an annular path which contains at least one member which is free to move therearound but is otherwise constrained to remain therewithin.

WO 01/66212 A1

Title: BALANCING DEVICE IN COMBINATION WITH CENTRIFUGAL EVAPORATOR

Field of invention

This invention concerns centrifugal evaporators and in particular an improved evaporator which is more versatile in use than such apparatus has been hitherto.

Background

Centrifugal evaporation is an effective way of evaporating solvents and separating out solid material for analysis and assessment. For various reasons, centrifugal evaporators have tended to be used in a batch mode that is, once loaded, they cannot normally be interfered with until the end of the evaporation cycle for the samples forming the batch.

There are a number of reasons for this batch approach to evaporation. One reason lies in the fact that high speed rotation of any object can introduce serious out of balance forces if the loading on the rotating drive is not uniform.

Non-uniformity of loading can arise due to uneven evaporation of different samples during an evaporation process. Typically the samples are contained in test tubes which themselves are supported in holders and the holders are arranged around a carousel or drum which itself is then spun around the vertical axis. Minor variations of solvent level in different test tubes can normally be accommodated, but if the solvent in a large number of samples in one region of the carousel tends to evaporate at a faster rate than the solvent in other regions, particularly a region diametrically opposite to that containing the faster evaporating samples, a serious out of balance mass can result with very significant out of balance forces being generated as the carousel is rotated at high speed.

In order to minimise the temperature rise which can occur as the solvent evaporates and dry test tubes are subjected to a continuous heating process, it has been proposed to provide relatively massive holders into which the test tubes are fitted, typically formed from good

thermally conductive material such as aluminium so that slower drying samples which retain volumes of liquid within the test tube for longer than the faster evaporating samples, will continue to absorb the heat and maintain a relatively constant temperature within the mass of aluminium or other thermally conductive material so preventing overheating of more rapidly drying samples.

The use of massive aluminium sample holders also assists in reducing the mass variation caused by uneven evaporation since the mass of aluminium remains the same whether the test tubes contain liquid or not. Typically the mass of the aluminium is somewhat greater than the mass of the test tubes even when filled with liquid, and by carefully loading each set of test tubes for each batch and where possible anticipating which of the samples is likely to evaporate faster than others, and mixing the faster and slower evaporating samples around the carousel, so out of balance forces arising from uneven evaporation have tended to be accommodated within each batch on a trial and error basis and with experience, skilled operators can maintain a reasonable balance as between fast and slow evaporating samples and prevent excessive out of balance forces arising.

However once an evaporator has been loaded with a given batch, it cannot normally be interfered with and samples added or removed during the processing of the batch, and the evaporator is thus tied up for periods of time while each batch is processed. If there is only one person using the evaporator and their work programme is carefully planned, this represents little if any problem.

In a busy laboratory however, two or three chemists may have access to only one evaporator and synchronising their requirements for the evaporator is difficult if not sometimes impossible. Consequently there is a need for the conventional centrifugal evaporator to be adapted to enable random usage so that batches requiring a long period of evaporation can be interrupted by the workers wishing to introduce other samples into the evaporator which may take a much shorter period of time to completion, or simply need to be processed urgently or to allow work programme to be completed in a sensible period of time. By continuing to use relatively large masses of aluminium within the rotor/carousel and inserting the sample holding test tubes into cavities in the good thermally conductive material, temperature variation can be minimised as described.

This leaves imbalance forces due to variations in mass as samples are added or removed and quite different samples are evaporated at different rates.

It is an object of the present invention to provide a modified centrifugal evaporator which at least in part overcomes the out of balance problem created by random or open access usage such as in a multi-user environment in a laboratory.

Summary of the invention

According to a first aspect of the present invention, an automatic balancing device is fitted to the rotating carousel or rotor of a centrifugal evaporator for the purpose of balancing the load as the carousel/rotor is rotated during an evaporation process and the automatic balancing device is selected so as to be capable of accommodating significant out of balance forces such as can arise if a number of sample containing test tubes are added to, or removed from, the carousel/rotor whilst others are left in place without any regard to balancing the weight distribution around the carousel/rotor.

According to a second aspect of the present invention there is provided a rotor for use in a centrifugal evaporator, having a plurality of sample holder receiving means, and being fitted with an automatic balancing device, wherein the automatic balancing device reduces out of balance forces that arise from the uneven distribution of sample holders around the rotor as the rotor is rotated during an evaporation process.

Typically the auto-balancing device is mounted on the rotor/carousel.

Preferably a ball bearing out of balancer is employed, which comprises a housing defining an annular path which contains at least one member which is free to move therearound but is otherwise constrained to remain therewithin.

Alternative auto-balancing devices may be employed such as a pendulum balancer or a liquid balancer.

Since the solvents which are boiled off during the evaporation process may be corrosive, the auto-balancing device is preferably mounted within a protective housing so as to protect the component parts thereof from attack from the solvent vapours.

In order to prevent ingress as pressure variations occur within the evaporating chamber, the housing or enclosure within which the auto-balancing device is located is preferably hermetically sealed.

The housing or enclosure may be formed from stainless steel such as stainless steel 316 and where the housing is formed from two or more parts, sealing between the various parts is preferably obtained by using O-ring seals.

If strong acids such as hydrochloric acid are employed within the chamber, then preferably the housing or enclosure is formed form PTFE coating stainless steel or is formed form a material such as Hastelloy or other materials which resist acid attack.

Preferably the rotor is manufactured from solid aluminium with multiple positions for receiving sample holders.

By providing a large number of different positions for the sample holders, users of the apparatus can be encouraged to distribute their samples around the rotor in a general random manner so as to spread the mass around the rotor as evenly as possible.

Some evaporators incorporate so-called swing rotors in which the sample containing test tubes are carried in pivoting supports so that as the array of tubes is rotated, the tubes will elevate due to centrifugal forces and swing out from the generally vertical position they will occupy when at rest to a generally horizontal attitude when the rotor is spinning at high speed. Such an arrangement has the disadvantage that in the vertical mode, the test tubes reduce the radial space available around the rotor axis for an auto-balancing device for an auto-balancing device. Almost invariably, the capacity of any auto-balancing device to accommodate out of balance loads will be at least in part proportional to the radius at which the compensating masses can themselves be rotated about the rotor axis. Consequently

downwardly hanging test tubes at a relatively small radius from the rotor axis reduces the maximum size of the auto-balancing device.

According therefore to a further preferred feature of the invention, in a centrifugal evaporator which is adapted to allow open access or random loading and unloading of samples, the rotor is designed and constructed so as to support the sample holding test tubes at an acute angle such that the axes of the test tubes lie on the surface of one or more cones, the vertical axes of which are coincident and coincide with the axis of rotation of the rotor. In this way the lower closed ends of the tubes are displaced radially by a considerable distance away from the axis of rotation of the rotor, and a larger auto-balancing device can be fitted immediately below the part of the rotor which supports the upper ends of the test tubes.

Preferably each sample holder is generally elongate and has a long axis, and each sample holder receiving means supports a sample holder such that the long axis of the sample holder subtends an acute angle to a plane perpendicular to the axis of rotation of the rotor.

Preferably each sample holder is inclined towards the axis of rotation of the rotor.

Typically the sample holders all subtend the same acute angle.

Preferably the sample holder receiving means comprises a first deck and at least a second parallel deck above the first deck, each deck determining the plane relative to which the sample holders in that deck are angled.

Typically that part of the rotor is a relatively thin circular plate which may itself be apertured so as to reduce its mass and the auto-balancing device is attached to a downwardly extending cylindrical hub at the centre of the rotor, the hub being adapted to be keyed or otherwise secured to a shaft of a drive unit for rotating the rotor.

A preferred auto-balancing device which employs tungsten carbide spheres having a specific gravity of 14.95 when used with a centrifugal evaporator of the type manufactured and supplied by Genevac Ltd can produce a balance correction capability of 42,000 g*mm. Such

an out of balance correction is sufficient to offset the imbalance which is produced by 150mm of solvent plus test tubes, plus aluminium sample holder.

Additional correction forces can be obtained by using Tungsten balls which have a specific gravity of 19.3.

In general the samples are heated during rotation using infra-red radiation and one or more infra-red pyrometers may be mounted within the chamber containing the rotor for measuring the temperature to which the samples are raised. Infra-red lamps may be used as the source of infra-red radiation, and preferably shields are provided to prevent the radiation impinging directly on the pyrometer.

According to a particularly preferred feature of the invention, the parts of the centrifugal evaporator which are connected to the rotating parts are preferably supported on compliant mountings. That is, the rotor may advantageously be supported in the centrifugal evaporator on compliant mountings.

The centrifugal evaporator itself may advantageously be supported on compliant mountings.

Preferably the natural frequency of the system is well below the operational frequency of the rotor since if this is not the case, the auto-balancing device may not function correctly.
That is, the rotor preferably has a natural frequency of oscillation (in Hertz) which is much less than the full speed frequency of rotation of the rotor (in revolutions per second) during the evaporation process. Preferably the second and subsequent harmonics of the natural frequency of oscillation of the rotor are much greater than the full speed frequency of rotation of the rotor during the evaporation process.

In general the auto-balancing device must be mounted so as to be concentric with the rotational axis of the rotor.

Preferably the plane of the imbalance within the rotor and the plane of the raceway containing the balancing sphere within the auto-balancer, should be as close as possible, and more preferably still, substantially coplanar.

According to a third aspect of the invention there is provided a centrifugal evaporator fitted with a rotor in accordance with the second aspect of the invention.

According to a fourth aspect of the invention there is provided a method of reducing out of balance forces that arise from the uneven distribution of sample holders around a rotor in accordance with the second aspect of the invention of a centrifugal evaporator as the rotor is rotated during an evaporation process.

According to a fifth aspect of the invention there is provided a process of evaporating a plurality of samples in which sample holders containing samples are loaded initially and centrifuged, the rotor is stopped and some of the sample holders are removed and/or other sample holders are added before the rotor is spun again; wherein a force to compensate for different and uneven loading of the rotor by the samples and sample holders is generated by a balancing device which comprises a housing defining an annular path which contains at least one member which is free to move therearound but is otherwise constrained to remain therewithin.

The invention will now be described by way of example, with reference to the accompanying drawings, in which:

Figure 1 is a sectional view of a rotor showing the position of an auto-balancer, sample holders and tubes;

Figure 2 is an isometric view of an evaporation chamber containing a rotor with 8 sample holders;

Figure 3 is a plot showing frequency response of an evaporator such as shown in Figures 1 and 2;

Figure 4 is a plot showing the performance of a typical auto-balancer compared with a normal rotor;

Figure 5 is an isometric view of a typically auto-balancing device incorporating an incomplete ring of spherical masses such as ball bearings which will move around the race to

occupy the position which gives maximum balancing force and minimum out of balance moments during rotation;

Figure 6 is a cross-section through an auto-balancer which may be fitted to the motor of an evaporator such as shown in Figures 1 and 2; and

Figure 7 is the right hand end of the cross-section shown in Figure 6 to an enlarged scale.

In Figure 1, test tubes such as 10 are shown mounted in sample holders such as 12 which themselves are received in pockets in a solid aluminium rotor 14 all contained within a generally cylindrical chamber 16 and carried by a rotatable shaft 18 supported in bearings 20 and 22 at its upper and lower ends respectively, the bearings being carried within a cylindrical sleeve 24 which extends centrally within the chamber 16 and is secured by means of a flange 26 to an opening in the lower end of the chamber 16. A drive connection to the shaft 18 is provided by means of a stub-shaft 28 and a motor or other driving device with or without a gearbox may be mounted to the underside of the chamber 16 (not shown) for rotating the shaft 18 and therefore the aluminium rotor 14 with the tubes and sample holders mounted therein.

Heat may be supplied by means of infra-red radiation from a source mounted in the wall of the chamber at 30 or projected through a window at 30, and an infra-red pyrometer 32 provides information on the temperature to which the aluminium rotor is raised during the evaporation process.

Although not shown, a cover is provided which is sealingly engaged to the upper end of the chamber 16 so as to form an enclosure within which the evaporation process can proceed.

The cover (not shown) is removed to give access to the sample holders and to allow tubes to be removed and placed in position as desired. To this end the flanged upper end of the chamber 16 includes a circular opening 34, the bounding flange 36 providing the means for securing thereto the cover.

Figure 2 is an isometric view which allows the test tubes such as 10 to be seen through the circular opening 34 in the upper flanged end of the chamber 36.

Figures 3 and 4 are self-explanatory and are the test results using auto-balancing devices on a centrifugal evaporator such as shown in Figures 1 and 2.

A perspective view of a typical auto-balancing device is shown in Figure 5 and this comprises a shallow cylindrical dish 38 within which a plurality of dense material ball bearings 40 are located around a central frusto-conical protrusion 42. The open upper surface of the cylindrical housing 38 is covered by means of a cover plate so as to form an enclosure such as shown at 42 in Figure 5. The housing parts are preferably hermetically sealed and as described may be formed from any suitable material given that the ball bearings are probably made from an active metal which will react with many of the solvent vapours given off by the evaporation process. Stainless steel, PTFE coated stainless steel and other materials may be employed for the housing of the auto-balancing device.

The latter is preferably mounted immediately below the rotor 14 and this is shown in Figure 1 at 44.

Figure 6 is a cross-section through that part of the rotor show in Figure 1 and illustrates the auto-balancing device which has been fitted to the evaporator. One of the balls 40 is shown in Figure 6, whereas the diametrically opposite region exposed by the cross-sectional view is shown at 46 to be devoid of any ball bearings.

O-ring seals 48 and 50 prevent the ingress of liquid or vapour to reach the annular region containing the ball bearings and the two parts of the housing which are identified by reference numerals 52 and 54 are secured together by means of rivets or screws and are typically formed from stainless steel 316.

In the device shown in Figure 1, nine Tungsten balls are located within the ball race which is capable of accommodating more than 9 balls. In order to accommodate the wear which could occur within such a device, a hardened steel raceway is provided at 56 as shown in the enlarged sectional view in Figure 7.

CLAIMS

1. A rotor for use in a centrifugal evaporator, having a plurality of sample holder receiving means, and being fitted with an automatic balancing device, wherein the automatic balancing device reduces out of balance forces that arise from the uneven distribution of sample holders around the rotor as the rotor is rotated during an evaporation process.

2. A rotor according to claim 1, wherein the automatic balancing device comprises a housing defining an annular path which contains at least one member which is free to move therearound but is otherwise constrained to remain therewithin.

3. A rotor according to claim 1, wherein the automatic balancing device comprises a pendulum balancer.

4. A rotor according to claim 1, wherein the automatic balancing device comprises a liquid balancer.

5. A rotor according to any preceding claim, further comprising a protective housing for the automatic balancing device to protect component parts thereof from attack by liquid spillage and/or solvent vapours from the sample holders.

6. A rotor according to claim 5, wherein the protective housing is formed from two or more parts.

7. A rotor according to claim 6, wherein sealing between the two or more parts is obtained by using O-ring seals.

8. A rotor according to claim 5 or 6, wherein the protective housing is hermetically sealed.

9. A rotor according to any one of claims 5 to 8, wherein the protective housing is formed from an acid-resistant material.

10. A rotor according to any one of claims 5 to 9, wherein the protective housing is formed from stainless steel.

11. A rotor according to claim 10, wherein the housing material is stainless steel 316.

12. A rotor according to any one of claims 5 to 9, wherein the protective housing is formed from Hastelloy.

13. A rotor according to any one of claims 5 to 12, wherein the protective housing is coated with PTFE.

14. A rotor according to any preceding claim, wherein the sample holder receiving means comprises or is substantially formed from aluminium.

15. A rotor according to any preceding claim, wherein each sample holder is generally elongate and has a long axis, and each sample holder receiving means supports a sample holder such that the long axis of the sample holder subtends an acute angle to a plane perpendicular to the axis of rotation of the rotor.

16. A rotor according to claim 15, wherein each sample holder is inclined towards the axis of rotation of the rotor.

17. A rotor according to claim 15 or 16, wherein the sample holders all subtend the same acute angle.

18. A rotor according to claim 15, 16 or 17, wherein the sample holder receiving means comprises a first deck and at least a second parallel deck above the first deck, each deck determining the plane relative to which the sample holders in that deck are angled.

19. A rotor according to any of claims 15 to 18, wherein the sample holder receiving means comprises upper and lower parts, the upper and lower parts having apertures for respectively supporting the upper, open ends, and the lower, closed ends, of the sample holders.

20. A rotor according to claim 19, wherein the automatic balancing device is fitted immediately below the upper part of the rotor.

21. A rotor according to claim 19 or 20, wherein the upper part comprises a circular plate.

22. A rotor according to any of claims 19 to 21, wherein the upper part has additional apertures to reduce its mass.

23. A rotor according to any preceding claim, further comprising a central downwardly extending cylindrical hub adapted to be secured to a shaft of a drive unit for rotating the rotor.

24. A rotor according to any of claims 2 and 5 to 23, wherein the at least one member is substantially spherical.

25. A rotor according to claim 24, wherein each member is formed from tungsten carbide.

26. A rotor according to claim 24, wherein each member is formed from tungsten.

27. A rotor according to any preceding claim, wherein the automatic balancing device is mounted coaxially with the rotational axis of the rotor.

28. A rotor according to claim 2 or any claim dependent therefrom, wherein the plane of the imbalance of the rotor and the plane of the raceway containing the at least one member are arranged so as to be substantially coplanar.

29. A centrifugal evaporator when fitted with a rotor as claimed in any of the preceding claims.

30. A centrifugal evaporator according to claim 29, wherein the rotor is supported on compliant mountings.

31. A centrifugal evaporator according to claim 29 or 30, supported on compliant mountings.

32. A centrifugal evaporator according to any of claims 29 to 31, wherein the rotor has a natural frequency of oscillation which is much smaller than the full speed frequency of rotation of the rotor during the evaporation process.

33. A centrifugal evaporator according to claim 32, wherein the frequencies of the second and subsequent harmonics of the natural frequency of oscillation of the rotor are much greater than the full speed frequency of rotation of the rotor.

34. A rotor for use in a centrifugal evaporator, fitted with an automatic balancing device and constructed, arranged and adapted to operate substantially as hereinbefore described by way of example with reference to, and as illustrated in, Figures 1 and 2 of the accompanying drawings.

35. A centrifugal evaporator substantially as hereinbefore described by way of example with reference to, and as illustrated in, Figure 1 of the accompanying drawings.

36. A method of reducing out of balance forces that arise from the uneven distribution of sample holders around the rotor of a centrifugal evaporator as the rotor is rotated during an evaporation process, wherein the rotor is as claimed in any of claims 1 to 34.

37. A method of reducing out of balance forces that arise from the uneven distribution of sample holders around the rotor of a centrifugal evaporator as the rotor is rotated during an evaporation process, wherein the rotor comprises an automatic balancing device as

hereinbefore described by way of example with reference to, and as illustrated in, Figures 1, 5, 6 and 7.

38. A process of evaporating a plurality of samples in which sample holders containing samples are loaded initially and centrifuged, the rotor is stopped and some of the sample holders are removed and/or other sample holders are added before the rotor is spun again; wherein a force to compensate for different and uneven loading of the rotor by the samples and sample holders is generated by a balancing device which comprises a housing defining an annular path which contains at least one member which is free to move therearound but is otherwise constrained to remain therewithin.

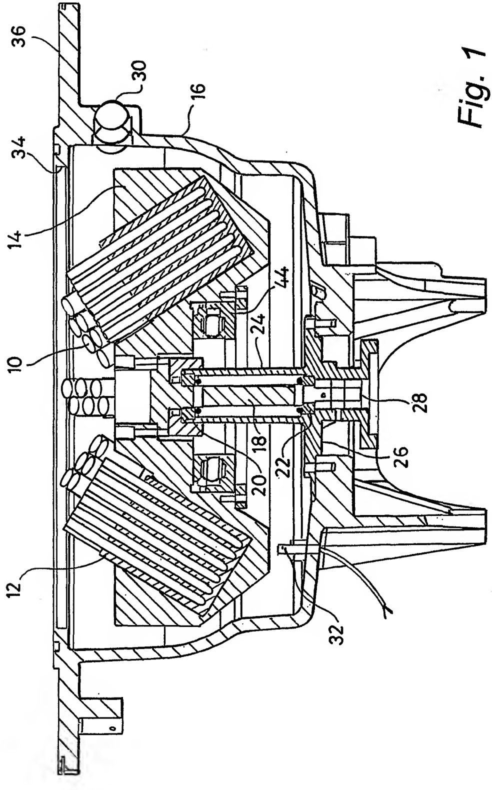



Fig. 1

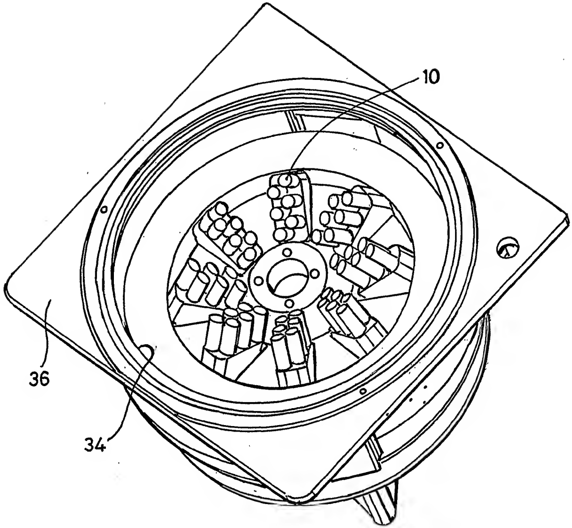



Fig. 2

**Autobalancer Trials 7-10-99**

Displacement (um)(200um range) vs Speed Controller Hz

20 g imbalance, water as 16-9-99, Rotor top level, Top sensor, with Autobalancer

*Lower (bench top) rubber feet isolated*

| Hz | um | RPM |
|---|---|---|
| 0 | | |
| 5 | 0.3 | 72 |
| 10 | 17 | 110 |
| 11 | 76 | 297 |
| 11.5 | 197 | 222 |
| 12 | 117 | 323 |
| 12.5 | 70 | |
| 13 | 53 | |
| 15 | 36 | 409 |
| 20 | 27 | 546 |
| 25 | 18 | 632 |
| 30 | 26 | 815 |
| 35 | 22 | 945 |
| 40 | 47 | 1076 |
| 45 | 35 | 1205 |
| 50 | 40 | 1331 |
| 55 | 76 | 1449 |
| 60 | 83 | 1514 |
| 65 | 86 | 1623 |
| 70 | 89 | 1654 |
| 75 | 89 | 1648 |
| 80 | 87 | 1608 |

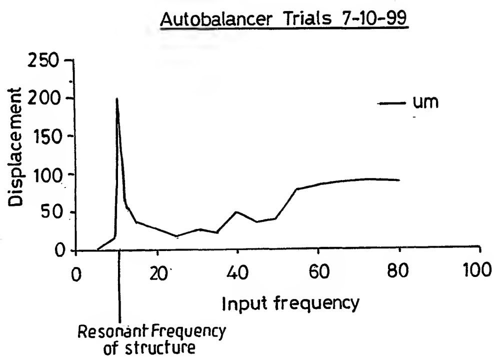
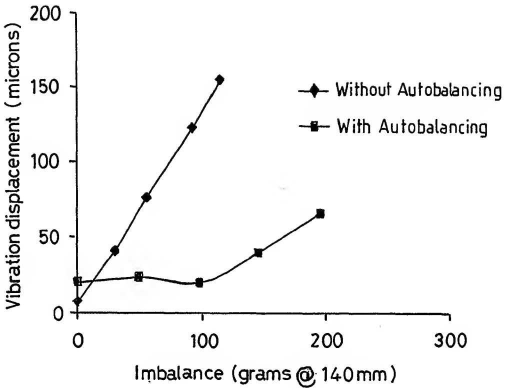



Fig. 3



Vibration displacement (microns)
200
150
100
50
0
0
100
200
300
Imbalance (grams @ 140mm)
Without Autobalancing
With Autobalancing


Fig. 4

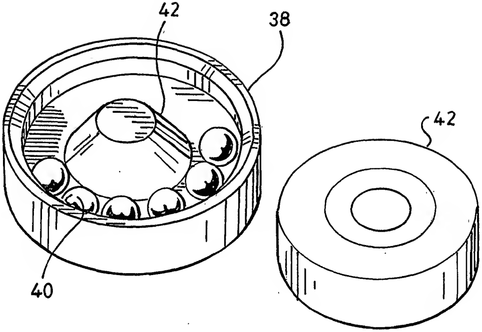



Fig. 5

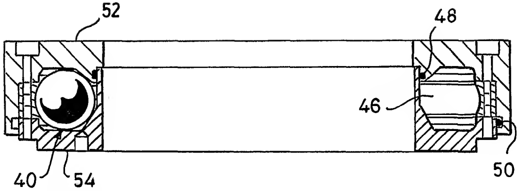



Fig. 6

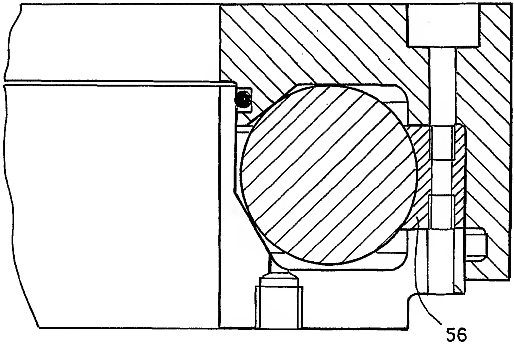



Fig. 7

# INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 01/00923

**A. CLASSIFICATION OF SUBJECT MATTER**
IPC 7 B01D1/22 B01D1/00 B04B9/14 F16F15/32

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01D B04B F16F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category ° | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X,P | WO 00 29122 A (COLE MICHAEL) 25 May 2000 (2000-05-25) page 1, line 1 -page 3, line 13; figures | 1,2, 5-17,23, 24, 26-29, 36,38 |
| X | DE 197 49 357 A (HITACHI KOKI KK) 25 June 1998 (1998-06-25) | 1,2,5,6, 15-17, 23,24, 27,28,34 |
| Y | column 1, line 3-16; figures | 18-21, 29-31, 35-38 |

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 26 April 2001 | 07/05/2001 |

| Name and mailing address of the ISA | Authorized officer |
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| European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016 | Persichini, C |

Form PCT/ISA/210 (second sheet) (July 1992)

Inte ial Application No
PCT/GB 01/00923

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category ° | Citation of document, with indication,where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X | US 3 692 236 A (LIVSHITZ STANLEY L ET AL) 19 September 1972 (1972-09-19) | 1,3, 15-17,23 |
| Y | column 1, line 5 -column 2, line 18; figures | 29-31, 36,38 |
| Y | US 5 084 133 A (GUY JEAN L ET AL) 28 January 1992 (1992-01-28) abstract; figures | 18-21, 29-31, 35-38 |
| Y | US 5 334 130 A (GLATER MICHAEL ET AL) 2 August 1994 (1994-08-02) abstract; figures | 29-31, 35-38 |

# INTERNATIONAL SEARCH REPORT

**ɔrmation on patent family members**

Inte ıal Application No

PCT/GB 01/00923

| Patent document cited in search report | | Publication date | Patent family member(s) | | Publication date |
|---|---|---|---|---|---|
| WO 0029122 | A | 25-05-2000 | GB | 2345655 A | 19-07-2000 |
| DE 19749357 | A | 25-06-1998 | JP | 10180147 A | 07-07-1998 |
| | | | US | 6132354 A | 17-10-2000 |
| US 3692236 | A | 19-09-1972 | AU | 3333171 A | 15-03-1973 |
| | | | BE | 773364 A | 30-03-1972 |
| | | | CA | 936506 A | 06-11-1973 |
| | | | CH | 542658 A | 15-10-1973 |
| | | | DE | 2152840 A | 31-05-1972 |
| | | | FR | 2110116 A | 26-05-1972 |
| | | | GB | 1346115 A | 06-02-1974 |
| | | | IT | 938889 B | 10-02-1973 |
| | | | NL | 7114176 A | 03-05-1972 |
| | | | SU | 463254 A | 05-03-1975 |
| US 5084133 | A | 28-01-1992 | FR | 2644356 A | 21-09-1990 |
| | | | CH | 682458 A | 30-09-1993 |
| | | | DE | 4008945 A | 27-09-1990 |
| | | | GB | 2230203 A,B | 17-10-1990 |
| | | | IT | 1240608 B | 17-12-1993 |
| | | | US | 5217572 A | 08-06-1993 |
| US 5334130 | A | 02-08-1994 | NONE | | |